

particularly pointed out in the appended claims, it being understood that various changes in the details may be made without departing from the spirit, or sacrificing any of the advantages of the present invention.

BRIEF DESCRIPTION OF THE DRAWINGS

For the purpose of facilitating an understanding of the invention, there is illustrated in the accompanying drawings a preferred embodiment thereof, from an inspection of which, when considered in connection with the following description, the invention, its construction and operation, and many of its advantages should be readily understood and appreciated.

FIGURE 1 is a schematic illustration of a first embodiment of the invention;

Fig. 2 is a schematic illustration of another embodiment of the present invention; and

Fig. 3 is a schematic illustration of another embodiment of the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring now to the drawings and more particularly to Fig. 1, there is shown a separation system 10 in which a vessel 15 has a generally cylindrical portion 16 with a dome shaped top 17 and a frustoconical shaped bottom 18 and exit pipe 19 extending from the bottom of the vessel 15. A reactor 20 of the type disclosed in the above-referenced patents has a outer liquid metal or sodium tube 21 and an inner halide vapor or titanium tetrachloride tube 22. A liquid metal or sodium supply tank 25 feeds sodium to the sodium or other liquid metal to the reactor 20 and a halide boiler 26 feeds the appropriate halide vapor to the reactor 20, all as previously described.

Internally of the vessel 15 is a downwardly sloping baffle 28 having a distal end 28a extending at a more acute angle and generally opposite to a sodium or liquid metal outlet 29. The liquid metal outlet 29 is in fluid communication with a metal or sodium pump 31 which leads to a heat exchanger 33 having a fluid inlet 34 and a fluid outlet 35. A liquid metal make-up line 37 32 is in communication with the supply tank or reservoir 25. A vent line 38 is provided in the tank or reservoir 25, as is well known in the engineering art.

A valve 40 with an actuator 41 is positioned in the exit 19 of the vessel 15 which is in communication with two exit lines 42 and 43, each of which being provided with a valve such as a valve 44 illustrated in line 42.

A filter assembly 45 includes a container 46 and a sloping filtered plate 47 for a purpose hereinafter set forth. A passivating gas inlet 50 has a valve 51 intermediate the source of passivating gas (not shown) and the container 46. A vacuum drying line 52 exits the container 46 and is provided with a valve 53. A slurry outlet line 56 at the bottom of the container 46 is provided with a valve 57 and a salt outlet line 61 is provided with a valve 62. Finally, a water wash inlet pipe 66 is provided with a valve 57 67.

The separation system 10 operates in following manner wherein material such as a metal or metal alloy is produced in the reactor 20 by the method previously described in the aforementioned and incorporated Armstrong patents. By way of illustration only, titanium or a titanium alloy may be made by the reduction of titanium tetrachloride vapor or a plurality of halide vapors for an alloy by an alkali or alkaline earth metal such as sodium or magnesium. Alloys are easily made with the Armstrong Process by mixing the halide vapors in the appropriate quantities and reducing them in the exact same manner as hereinbefore described. In any event, using a large excess of the reducing metal to control the reaction produces a slurry of excess reducing metal, such as sodium, the metal particulates such as titanium and another reaction product such as salt particles, sodium chloride. The slurry leaving the reactor 20 may be at a variety of temperatures controlled, in one instance, by the amount of excess reducing metal present.

In an actual example, the slurry may typically have up to about 10% by weight particulates, and the particulates may be salt having diameters on average of from about 10 to about 50 microns and titanium having diameters on average in the range of from about 0.1 micron to about 500 microns, the titanium particulates or powder may be more likely to be in the range of from about 1-10 microns and the agglomerated ligaments (lumps) of the titanium in the range of between about 50 and about 1000 microns. This combination of liquid metal, salt particulates and titanium particulates leave the nozzle reactor 20 and enter the vessel 15. The salt in the

vessel 15 is indicated to be at a level of which may be arbitrarily chosen so long as it is below the sodium outlet 29. The salt may be the reaction product salt, for instance sodium chloride, or a salt mixture which has a melting point lower than the reaction product salt. Although the salt may be as stated any salt, preferably the salt is the product of reaction or a mixture thereof, for instance an eutectic such as the calcium chloride- sodium chloride eutectic which melts at about 600°C.

The entire system 10 then may be operated at a lower temperature. For instance, sodium chloride melts at about 850°C. so if the salt in the vessel 15 is sodium chloride, then the vessel 15 must be operated above the melting point thereof, but as the eutectic melts at 600°C, this reduces the operating temperature. In any event, irrespective of what salt is present at the level 30 in the vessel 15, the liquid metal will float due to density differences and be extracted through the outlet 29 by means of the sodium or liquid metal pump 31. A heat exchanger 33 having suitable inlet and outlet lines 34, 35 serves to reduce the temperature of the sodium out from the 600° in the vessel 15 (by way of example only) so that the recycled sodium enters the reactor 20 at a preselected temperature (for instance about 400°C). The baffle 28 and 28a prevents particulates entering the vessel 15 from the reactor 20 from being sucked into the sodium outlet 29.

As particulates settle in the lower portion 18 of the vessel 15, the particulate concentration is increased due to the removal of sodium through the line 29. Upon actuation of the valve 40, concentrated slurry will drain through the outlet or exit 19 through line 42 into the filter assembly 45. In the filter assembly 45, which is maintained by temperatures sufficient to keep the molten salt in a liquid phase, metal particles collect on the filter plate 37 47 while salt passing through the filter plate exits through line 61 to be returned, for instance, to an electrolytic cell (not shown). The valve 62 opens the line 61 to permit the salt to drain while valve 57 is closed to prevent material from exiting the filter assembly 45. After a sufficient filter cake has been built up, the valve 62 is closed, the valve 44 is closed and the vacuum drying line 53 is opened after the filter cake has cooled to less than about 100 °C so that the passivating gas which may be argon and a small percentage of oxygen may be introduced into the container 46 by actuation of the valve 51. After the filter cake

which may be principally titanium powder with some salt is passivated, then the valve 51 is closed and the water wash valve 67 opened thereby allowing water to enter into the container 46 which both dissolves salt and moves the filter cake through line 56 to a finish wash and classification, it being understood that valve 67 will be opened prior to the water wash. The salt coming out of the filter assembly 45 through line 61 can be recirculated to the vessel 15 as indicated by the line 61a.

As seen therefore, the separation system 10 depends on the difference in gravity between the unwanted liquid metal constituent of the slurry and the salt and metal particulates produced during the reaction of the dried vapor and the reducing metal. Although this separation system 10 is a batch system, it can be rapidly cycled from one filter assembly 45 to other filter assemblies as needed through a simple valve distribution system, as is well known in the art.

Although the above example was illustrated with sodium and titanium tetrachloride, it should be understood that any material made by the Armstrong Process may be separated in the aforesaid manner.

Figure 2 shows an alternate embodiment separation system 80 in which a vessel 85 is similar to the vessel 15 and has a cylindrical portion 86, a dome top 87 and a frustoconical bottom 88 having an exit 89 88 extending therefrom. A reactor 90 of the same type as hereinbefore described is in communication with the vessel 85 and has a halide inlet 91 and a reducing metal inlet 92. A slurry outlet 93 which is in communication with the top 87 of the vessel 85. The filter 95 is any suitable filter, well known in the art, but preferably, for purposes of illustration only, is a "wedge screen filter" of a size to pass up to 125 micron particles. The material that flows through the filter 95 exits the vessel 85 through an output line 96 and flows into a gravity separator 97. The gravity separator 97 is frustoconical in shape and has an outlet 99 through which the heavier of the materials flows, in this particular case sodium chloride. An outlet 98 takes the lighter of the material, in this case sodium and recycles same through appropriate filters and other mechanisms, not shown, to the reactor 90. In this embodiment, the vessel 85 is maintained at an elevated temperature of about 850°C with either internal or external heaters, as is well known in the art, in order that the salt in this case, sodium chloride, is liquid or molten. The